Nitrate-Nitrite, Manual Cadmium Reduction **SM 18th, 19th, 20th Ed 4500-NO_3-E**

ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.

Facility Name:_____VELAP ID_____

Assessor Name:_____ Analyst Name:_____ Inspection Date_____

Relevant Aspect of Standards	Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
1) If analyzing for Nitrite **OR** Nitrate, are samples preserved in the following manner: Nonpotable: ≤ 6°C, analyzed within 48 hours of collection Drinking water: ≤ 6°C, analyzed within 48 hours unless chlorinated, for which nitrate can be held up to 14 days	40 CFR 136.3 Table II, 40CFR 141.23 (k)(2), EPA 815-R-05-004				
2) If analyzing for Nitrate-Nitrite, are samples preserved to pH <2 with sulfuric acid, preserved by storing at ≤6°C, and analyzed within 28 days of collection?	40 CFR 136.3 Table II, 40CFR 141.23 (k)(2)				
3) Were unchlorinated samples preserved with 2 mL concentrated H_2SO_4/L if stored for longer than 2 days?	NO_3^- A Intro				
4) When samples were preserved with acid, was the determination of NO_2^- not needed?	NO_3^- A Intro				
5) Were turbid samples filtered prior to analysis?	NO_3^- A Intro				
6) Were samples where copper, iron, and other metal concentrations above several mg/L was suspected treated with EDTA prior to analysis?	4500-NO_3^- E 1 b				
7) Where samples were contaminated by oil and grease, were they solvent extracted prior to analysis?	4500-NO_3^- E 1 b				
8) Were samples checked for residual chlorine, and, if RC was present, were samples treated with sodium thiosulfate?	4500-NO_3^- E 1 b				
9) If a spectrophotometer was used, did it have a wavelength of 543 nm with a path length of 1 cm or longer?	4500-NO_3^- E 2 b 1				
Notes/Comments:					

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10) If a filter photometer was used, did it have a light path of 1 cm or longer and a transmittance near 540 nm?	4500-NO_3^- E 2 b 2				
11) Did absorbances of reagent blanks never exceed 0.01?	4500-NO_3^- E 3 a				
12) Were 25 g aliquots of 20 to 100 mesh Cd granules washed with 6N HCl?	4500-NO_3^- E 3 b				
13) In repeated cycles were Cd granules next swirled with 2% $CuSO_4$ solution for 5 minutes or until blue color faded until a brown colloidal precipitate developed?	4500-NO_3^- E 3 b				
14) Was an EDTA solution prepared by dissolving 13 g NH_3Cl and 1.7 g disodium ethylenediamine tetraacetate in 1 L water and then adjusting the pH to 8.5?	4500-NO_3^- E 3 d				
15) Was an color reagent prepared by diluting 100 mL 85% phosphate acid and 10 g sulfanilamide and 1 g N-(1-naphthyl)-ethylenediamine to 1 L with reagent water?	4500-NO_3^- E 3 c				
16) Was an ammonium chloride-EDTA solution prepared by diluting 300 mL of the above ammonium chloride-EDTA solution to 500 mL with water?	4500-NO_3^- E 3 d				
17) Was a copper sulfate solution prepared by diluting 20g $CuSO_4 \bullet 5H_2O$ to 1 L with water?	4500-NO_3^- E 3 g				
18) Was a stock nitrate solution prepared by drying KNO_3 at 105°C for 24 hours and diluting 0.7218 g to 1000 mL with H_20 and then preserving it with 2 mL of $CHCl_3$/L?	4500-NO_3^- E 3 h				
19) Was an intermediate nitrate solution prepared by diluting 100 mL of above nitrate solution to 1000 mL with water and preserved with 2 mL of $CHCl_3$/L?	4500-NO_3^- E 3 i				
20) Were the nitrate solutions not used for longer than 6 months?	4500-NO_3^- E 3 h				
21) Was a stock nitrite solution prepared by diluting 1.232 g $NaNO_2$ with water and preserving with 1 mL $CHCl_3$ and then titrating it to determine its concentration?	4500-NO_3^- E 3 j				
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22) Was an intermediate nitrite solution prepared by diluting the volume of above solution necessary to make 50 µg N/ 1.00 mL?	4500-NO_3^- E 3 k				
23) Was the intermediate nitrite solution prepared daily?	4500-NO_3^- E 3 k				
24) Was a working nitrite solution prepared by diluting 50.0 mL to 500 mL with water?	4500-NO_3^- E 3 l				
25) Did reduction columns first have glass wool plugs inserted into them, then get filled with water, and then have 18.5 cm of Cu-Cd granules added to them?	4500-NO_3^- E 4 a				
26) Were reduction columns prepped by washing with 200 mL of dilute NH_4Cl-EDTA then about 100 mL of 25% 1.0 mg NO_3^-N/L+75% NH_4Cl-EDTA?	4500-NO_3^- E 4 a				
27) Were sample pHs adjusted to be between 7 and 9 with dilute HCl or NaOH?	4500-NO_3^- E 4 b 2				
28) Were 25 mL sample volumes mixed with 75 mL volumes of NH_4Cl-EDTA solution?	4500-NO_3^- E 4 b 3				
29) Were the first 25 mL of sample NH_4Cl-EDTA solution mixtures that passed through the column discarded?	4500-NO_3^- E 4 b 3				
30) If columns were used for more than several hours, were they stored with dilute NH_4Cl-EDTA solution and not allowed to dry?	4500-NO_3^- E 4 b 3				
31) Was color reagent addition never more than 15 minutes after reduction?	4500-NO_3^- E 4 b 4				
32) Were 2.0 mL of color reagent added to 50 mL volumes of reduced sample mixtures, and the absorbances measured against DI blanks at 543 nm?	4500-NO_3^- E 4 b 4				
33) Were the absorbances of reduced sample mixtures measured between 10 minutes and 2 hours after color reagent addition?	4500-NO_3^- E 4 b 4				
34) Was at least one NO2- standard compared to a reduced NO3- standard at the same concentration to verify column efficiency?	4500-NO_3^- E 4 c				
35) Were columns reactivated when above column efficiency dropped below 75%?	4500-NO_3^- E 4 c				
Notes/Comments:					